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IS 7559 (1992): Salicylic Acid, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS 7559 : 1992

(Reaffirmed 2004)

भारतीय मानक

सैलिसिलिक एसिड, तकनीकी – विशिष्ट

(पहला पुनरीक्षण)

Indian Standard

SALICYLIC ACID, TECHNICAL –
SPECIFICATION

(*First Revision*)

UDC 547.587.11

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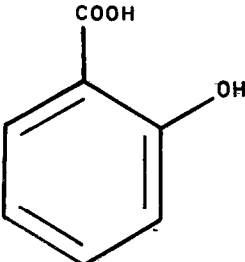
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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Salicylic acid is used as a preservative and in the manufacture of dyes and perfumes. It is also used in preventing pre-curing in rubber compounds and as an organic intermediate. It is also known as ortho-hydroxybenzoic acid. Its molecular, structural formula and molecular mass are given below:

<i>Molecular Formula</i>	<i>Structural Formula</i>	<i>Molecular Mass</i>
$C_6H_4(OH)(COOH)$		138.12

This standard was published in 1974. The Committee decided to revise it in view of the improved quality of the material which is currently being made available in the country. In this revision the requirement of moisture content, residue on ignition and melting range have been revised. Thin layer chromatographic method has been introduced for estimation of impurity in the material.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SALICYLIC ACID, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for salicylic acid technical.

2 REFERENCES

The following Indian Standards are necessary adjunct to this standard:

IS No.	Title
323 : 1959	Rectified spirit (<i>revised</i>)
1070 : 1977	Water for general laboratory use (<i>second revision</i>)
4905 : 1968	Methods of random sampling
5762 : 1970	Methods for determination of melting range temperature

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white to pale powder.

3.2 Identification Test

The material shall pass the tests as prescribed in Annex A.

3.3 The material shall also comply with the requirements given in Table 1, when tested according to the methods referred in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in polythene bags bottle or fibre drums or as agreed to between the purchaser and the supplier.

4.2 Marking

The containers shall be marked with the following information:

a) Name of the material;

- b) Indication of the source of manufacture;
- c) Net mass; and
- d) Lot or batch number, in code or otherwise.

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

The procedure for sampling and criteria for conformity of the material shall be as prescribed in Annex F.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods referred in 3.2 and in col 4 of Table 1.

6.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (see IS 1070 : 1977) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Salicylic Acid, Technical

(Clause 3.3)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity (as C_6H_4OH . $COOH$), percent by mass (on dry basis) <i>Min</i>	98.5	Annex B
ii)	Moisture, percent by mass, <i>Max</i>	0.35	Annex C
iii)	Residue on ignition, percent by mass, <i>Max</i>	0.3	Annex D
iv)	Melting range, °C	158.0 to 161.0	IS 5762 : 1970
v)	<i>p</i> -hydroxybenzoic acid percent by mass, <i>Max</i>	0.5	Annex E

ANNEX A

(Clause 3.2)

IDENTIFICATION TESTS

A-1 The following identification tests shall be carried out.

A-1.1 Add dilute hydrochloric acid to a neutralized solution of the material. A crystalline precipitate of salicylic acid is formed which shall be soluble in aqueous ammonium acetate, solvent ether and chloroform.

A-1.2 Add ferric chloride solution to a neutralized solution of the material. An intense reddish violet colour will be produced.

A-1.3 Dissolve 25 g of the material in 100 ml of 10 percent caustic soda solution. A clear light yellowish brown liquid solution shall be produced.

ANNEX B

[Clause 3.3, Table 1, Sl No. (i)]

DETERMINATION OF PURITY

B-1 REAGENTS

B-1.1 Phenol Red Solution

Warm 0.05 g of phenol red (phenol-sulphonaphthalein) with 2.85 ml of 0.05 N sodium hydroxide solution and 5 ml of 90 percent ethanol; after solution is effected, add sufficient quantity of 20 percent ethanol to produce 250 ml.

B-1.2 Standard Sodium Hydroxide Solution — 0.5 N.

B-1.3 Rectified Spirit — conforming to IS 323 : 1959.

B-2 PRODEDURE

Weigh accurately about 2.0 g of the material, previously dried over silica gel for 3 hours, and transfer into a 250-ml conical flask. Dissolve

in 15 ml of warm rectified spirit, previously neutralized with phenol red solution. Add 20 ml of water and titrate with standard sodium hydroxide solution (keeping the contents of the flask always in dissolved state, by heating if necessary) using phenol red as indicator.

B-3 CALCULATON

Salicylic acid content,

$$\text{percent by mass} = \frac{13.812 \times V_1 \times N_1}{M}$$

where

V_1 = volume, in ml, of standard sodium hydroxide solution used;

N_1 = normality of standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

ANNEX C

[Clause 3.3, Table 1, Sl No. (ii)]

DETERMINATION OF MOISTURE

C-1 PROCEDURE

Weigh accurately about 10.0 g of the material in a watch glass, previously dried by heating at 100°C and weighed. Place the contents in a vacuum desiccator, with concentrated sulphuric acid as desiccant. Apply vacuum for 5 to 10 minutes. The watch glass and the contents are allowed to remain in desiccator for 16 hours. Open the tap of the desiccator very slowly, allowing air to enter without disturbing the material on the watch glass. Finally, weigh the material along with the watch glass.

C-2 CALCULATION

$$\text{Moisture, percent by mass} = \frac{(M_1 - M_2) \times 100}{M_1 - M}$$

where

M_1 = mass, in g, of the watch glass with material before drying;

M_2 = mass, in g, of the watch glass with the material after drying; and

M = mass, in g, of the empty watch glass.

ANNEX D

[Clause 3.3, Table 1, Sl No. (iii)]

DETERMINATION OF RESIDUE ON IGNITION

D-1 OUTLINE OF THE METHOD

A known quantity of the material is ignited at $600 \pm 25^\circ\text{C}$ and the residue cooled and weighed.

D-1.1 Apparatus

D-1.1.1 Basin — made of silica.

D-1.1.2 Furnace — capable of maintaining a temperature of $600 \pm 25^\circ\text{C}$.

D-1.2 Procedure

Gently volatilize 10.0 g of the material in successive small portions in the weighed basin and ignite the residue at $600 \pm 25^\circ\text{C}$. Cool in

a desiccator and weigh. Repeat ignition, cooling and weighing till constant mass is obtained.

D-1.3 Calculation

$$\text{Residue on ignition, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass, in g, of the residue after ignition; and

M = mass, in g, of the material taken for the test.

ANNEX E

[Clause 3.3, Table 1, Sl No. (v)]

ESTIMATION OF *p*-HYDROXYBENZOIC ACID

E-1 GENERAL

p-hydroxybenzoic acid in salicylic acid, technical is estimated by ascending thin layer chromatographic method.

E-1.1 Apparatus

E-1.1.1 Thin Layer Chromatographic Glass Plates — 20 cm × 20 cm.

E-1.1.2 Micropipettes — 10 µl capacity.

E-1.1.3 Chromatographic Chamber — 22 cm × 10 cm × 22 cm.

E-1.1.4 Adjustable Thin Plate Applicator

E-1.1.5 Chromatographic Spray Bottle

E-1.1.6 Weighing Bottle

E-1.2 Reagents

E-1.2.1 Sample Solution — 10 percent solution in methanol.

E-1.2.2 Reference Solution of *p*-Hydroxybenzoic Acid (99.9 percent) — 0.05 percent solution in methanol.

E-1.2.3 Silica gel G — Neutral.

E-1.2.4 Developing Solvent — Petroleum spirit (60–80°C) / Ethyl acetate/glacial acetic acid (80 : 10 : 5) (v/v).

E-1.2.5 Spray Solution — Dissolve 0.5 g of potassium permanganate in 50 ml of 1 N sodium hydroxide solution and add 50 ml of water.

E-1.3 Procedure

E-1.3.1 Preparation of Chromatographic Plates— Mix 50 g of natural silica gel G with about 90 ml of water for three minutes. Coat the plate with silica gel G by means of an applicator

so that the layer of coating is uniform and the thickness does not exceed 0.25 mm. Place the plate in the oven maintained at 110°C for 90 minutes. Mark a thin line, by means of a needle widthwise on all plates at about 3 cm from the upper edge. Remove a band of about 5 mm width silica gel G from either of the lateral sides of each plate.

E-1.3.2 Chromatographic Separation — Pipette out 10-µl of the sample solution **E-1.2.1** and 10 µl of reference solution (**E-1.2.2**) and apply in the form of a uniform line of about 14 cm length on the plate at a distance of 3 cm from the bottom edge. After complete evaporation of the solvent, place the plate in the chromatographic chamber and allow the developing solvent (**E-1.2.4**) in the chamber to migrate upto 13 cm. Remove the plate and let it dry in the air for about 30 minutes. Dry the plate completely in room temperature and spray with solution of potassium permanganate in 1 N sodium hydroxide solution. Let the plate stand for 5 minutes. The separated spots characteristic of constituents can be identified by their colour as under:

Content	Zone	R _f Value	Colour
Unknown	I	0.33	Yellow
<i>p</i> -hydroxybenzoic acid	II	0.39	Yellowish white
Main	III	0.80	Yellowish white

E-1.4 Reporting

Report the impurity of *p*-hydroxybenzoic acid as that which is nearest in intensity to the reference samples. In case the colour intensity does not come in the range of the standard spots of impurity, repeat the whole procedure using suitable concentration of various impurities.

ANNEX F

(Item 5)

SAMPLING OF SALICYLIC ACID, TECHNICAL

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

F-1.2 Sampling instruments shall be clean and dry.

F-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

F-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

F-1.5 The sample shall be placed in clean, dry and airtight glass containers or other suitable containers on which the material has no action.

F-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, batch number and other important particulars of the consignment.

F-1.8 Samples shall be stored in a cool and dry place.

F-2 SAMPLING INSTRUMENT

A stainless steel scoop of suitable dimensions shall be used to draw the samples.

F-3 SCALE OF SAMPLING

F-3.1 Lot

In a single consignment, all the containers of the same size, containing material from the same batch of manufacture, shall constitute a lot.

F-3.2 For ascertaining the conformity of the lot to the requirement of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected from the lot shall depend on the size (N) of the lot and shall be in accordance with Table 2.

Table 2 Scale of Sampling
(Clause F-3.2)

Lot Size	Number of Containers to be Selected
N	n
(1)	(2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 150	6
151 to 300	7
301 and above	8

F-3.3 These containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS 4905 : 1968 may be followed.

F-4 TEST SAMPLES AND REFEREE SAMPLE

F-4.1 From each of the containers selected according to F-3.2 a representative portion of the material shall be drawn. These samples shall constitute individual samples.

F-4.2 From each of these individual samples (F-4.1) approximately equal quantity of the material shall be drawn and thoroughly mixed to constitute a composite sample of not less than 500 g. The composite sample shall be transferred to a polythene bag and labelled with full identification particulars of the sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third as a referee sample.

F-4.2.1 The referee test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of any dispute.

F-5 TESTS

Tests for the determination of all characteristics given in 3 shall be carried out on the composite sample.

F-6 CRITERIA FOR CONFORMITY

For determining conformity of the lot to the requirements of this specification, test results on the composite sample shall meet the corresponding requirements given in 3.1, 3.2 and 3.3.

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The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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